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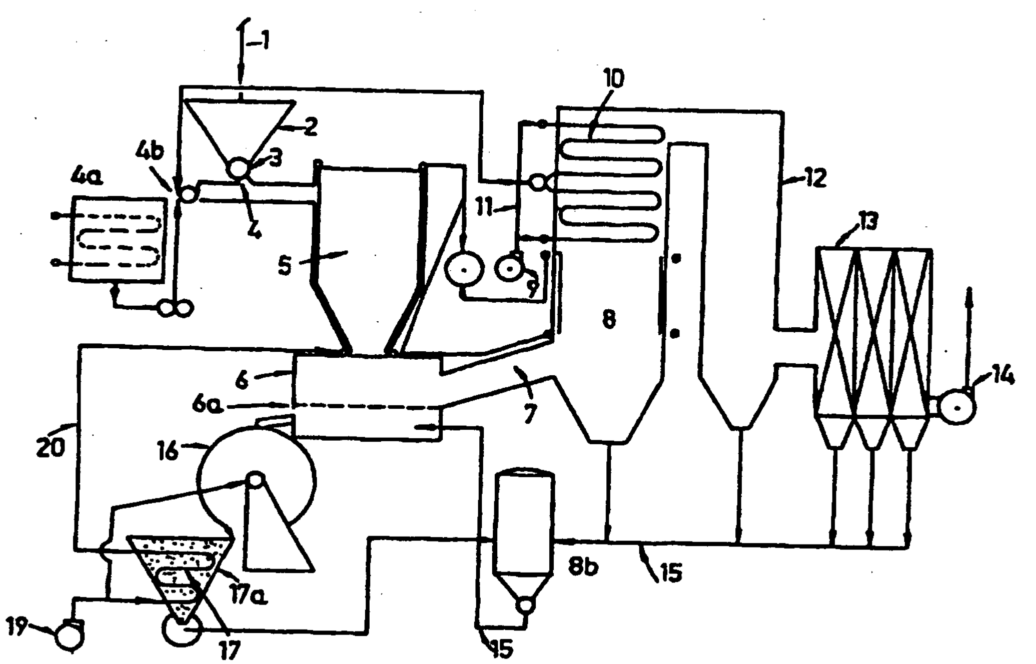

(57) Abstract

A process for producing granulated potassium chloride, comprising: forming a mixture of potassium sulfate and potassium chloride; melting said mixture (5); bringing said molten mixture into physical contact with a cooling surface provided with recesses (16), said recesses being substantially of dimension and shape of the granules to be produced, said surface being cooled, whereby solidification of the molten mixture is obtained; and collecting the solid granules from the said surface.

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PROCESS FOR THE PRODUCTION OF GRANULATED POTASSIUM CHLORIDE

This invention relates to the manufacture of inorganic fertilizers, and more particularly to the production of granulated potassium chloride.

Potassium chloride is an inorganic fertilizer component. It is used in granulated form and various processes are known in the art for preparing it in such a form. One such process consists in feeding particulate potassium chloride to calendering rollers, whereby to produce a sheet which is then ground to granules. However. this process has a low efficiency and only about 30% of the product is satisfactorily granulated by passage between calendering rollers. Further, a significant amount of the material (3-5%) is lost and the resistance of the granules to compression is relatively low, specifically they have a crushing load - the compression load which destroys the granules by crushing them - of about 2-2.5 kg.

Another method of producing potassium chloride granules involves preparing a mixture of the raw material with 3-10% of ammonium chloride. humidifying the mixture by the addition of water, passing it through a sieve and drying the resulting granules. This process. however. is relatively expensive. and does not permit the use of high temperatures and therefore necessarily comprises a relatively long drying phase.

It is also been attempted to increase the strength of the potassium chloride granules by the addition of secondary products formed in the manufacture of said chloride, particularly magnesium chloride in the amount of about 3.5% and calcium chloride in the amount of about 1.5%. In this way, however, a more hygroscopic material is produced and the necessity of using a relatively low drying temperatures (e.g., about 200°C) increases the duration of the drying and the expense of energy.

It is particularly difficult to obtain KCl granules having dimensions of about 3-4 mm and adequate compression strength, e.g. of 3 to 7 kg and such production is not possible by the methods and apparatus of the prior art.

It is a purpose of this invention to overcome the defects of the known processes and to provide a process for the production of granulated potassium chloride which produces the desired product in granulated form. without substantially increasing manufacturing costs.

It is another purpose of this invention to provide such a process which produces potassium chloride granules having a high resistance, in particular a resistance to compression or crushing load of 3 to 7 kg.

It is a further purpose of this invention to provide such a process which permits to produce granules having dimensions of 1.5 mm and more.

It is a further purpose of this invention to provide such a process which reduces the hygroscopicity of the final product.

It is a still further purpose of this invention to provide such a process which affords a saving in the energy required for carrying it out.

These and other purposes and advantages of the invention will appear as the description proceeds.

The process according to the invention comprises the steps of:

- forming a mixture of potassium sulfate and potassium chloride;
- melting said mixture;
- bringing said molten mixture into physical contact with a cooling surface provided with recesses, said recesses being substantially of dimension and shape of the granules to be produced. said surface being cooled, whereby solidification of the molten mixture is obtained: and
- collecting the solid granules from the said surface.

Preferably, the amount of K_2SO_4 used is about 1 to 5% by weight of the KCl-K_2SO_4 mixture.

Generally, the granules obtained from the solidification of the molten drops have sizes comprised between 1.5 and 6 mm.

The granulation process is characterized by a cooling method which was discovered to be very efficient. According to said method, the molten mixture is poured on a cooling surface having recesses thereon, the recesses substantially of dimension and shape of the granules to be produced. According to a preferred embodiment of the invention. said surface is provided

by a drum capable of rotational movement. Heat is transferred from the molten drops collected in the recesses to the substance of which the drum is made of (preferably iron), while the drum being continuously cooled by water. Granules are therefore obtained in the recesses, and may then be collected in a container wherein heat exchanger is placed to complete the cooling procedure, while the heat is recycled.

Preferably, the drum makes a continuous rotational movement, through which the granules obtained in the recesses fall to a container for collection, because due to the cooling process their dimensions become smaller than these of the recesses. Of course, any other convenient procedure which allows cycling process, such as conveyer belt, may be adapted, and further means to allow the falling of said granules from said recesses to said container may be added.

As stated, the surface provided with recesses thereon may be of any desired structure: the recesses, for example, may have a rectangular or hexagonal cross section, and their bottom is usually rounded. According to one embodiment of the invention, the drum is coated with a porous coating to provide a protection for the drum. According to another embodiment of the invention, a second roller may be coupled to the drum to break the material crystalized outside the recesses. By the use of said second roller, excess of material is removed from the recesses, leveling the upper face of the granules produced, and thus obtaining well defined shapes of the granules.

The apparatus according to the invention comprises

- means for forming a mixture of potassium sulfate and potassium chloride;
- means for bringing said mixture to an oven;

- means for melting said mixture in the oven;
- means for obtaining the crystals by bringing said molten mixture into a physical contact with a cooling surface provided with recesses, said recesses being substantially of dimension and shape of the granules to be produced, said surface being cooled, whereby solidification of the molten mixture is obtained; and
- means for collecting the solidified granules from the surface and further cooling them, and for recycling the heat.

In a preferred embodiment of the invention, means for improving the energy use are provided. in order to make the process more industrially attractive.

As may be clear to one skilled in the art, several ways for improving the consumption of energy may be applied in the above described process and apparatus, two of which are exemplified in the drawings.

In the drawings:

Fig. 1 illustrates one preferred embodiment of the invention.

Fig.2 illustrates another preferred embodiment of the invention based on the use of cyclone heat exchanger.

Fig. 3 shows a preferred embodiment of the drum and its cooling system.

Fig. 4 shows another preferred embodiment of the drum. when coupled to a second roller (a view of the rotary axis).

Referring to Fig. 1, the solid material to be treated, comprising a mixture of about 95 to 99% of KCl and 1 to 5% of K_2SO_4, indicated at (1), is charged into a container or like apparatus 2. By any convenient feeder (3), such as screw-feeder, the mixture is fed, in a controlled manner, to conduit (4). Numeral (4a) indicates a reservoir containing a fuel, such as mazout. A different fuel could, of course, be used. The preheated fuel is fed, together with preheated air (according to one embodiment of the invention, the hot air is obtained from heat exchanger (10), to be described hereinafter) to a burner, which is schematically indicated as 4b. The product of the burning of the fuel and the hot air, i.e., combustion gases. flow through conduit (4) and drive the salt mixture to melting oven (5). which is a cyclone oven. The salt mixture completely melts in oven (5) (at about 830-850 °C). Numeral (6) indicates a container in which the melted salt mixture is collected, and numeral (6a) stands for the level of the molten salt in said container. This mixture is now brought into contact with the granulation system. The granulation system is composed of a drum, (16). made of a material which is a good heat conductor. such as iron, capable of making a rotational movement. Water (20) is pumped by a pump (19), in order to cool the rotating drum. in a manner specifically shown in figure 3. The surface of said drum has recesses on, and the molten mixture is poured from container 6 to the recesses of said surface. Due to the rotational movement of the drum, crystals obtained in the recesses fall, being collected in a container (17a), in which heat exchanger (17) is placed. to further carry out the cooling process of the granules and to recycle the heat absorbed.

As stated. the apparatus described above may be further modified. in order to make it more attractive from the economical and industrial points of view. For

example, it may be coupled to another system. in order to improve the energy use and to avoid possible loses of heat and material. Particularly, the combustion gases have a temperature of about 880-950°C at their exit from oven 5, and in addition they carry some small amounts of the salt mixture in the form of very small drops or in the form of molecules which are in the gaseous phase. Thus, the suspension of molten salt drops in the combustion gases flows through gas conduit (7) into a gas separator (8), walls of which are cooled by water. KCl-K_2SO_4 particles. formed as a result of the cooling of the suspension, fall to the bottom of the separator and therefrom into a vessel (8b). A pneumatic system (15) is used to transfer it to the container (6), to be melted therein and further to be subjected to the granulation procedure. as described above. The gases are brought into contact with heat exchangers. Outside air (11) is driven from fan (9) to said heat exchanger, it is heated therein from ambient temperature to about 450°C. According to one preferred embodiment of the invention. this heated air may now be used in the burner (4b), for the combustion process. as described above. The cooled gases, together with the materials that have not separated from them, pass through a separator (12), and from it to mechanical filters (13), from which they are drawn by exhauster fan (14), while the salt granules settle to the bottom of said second separator (12) and said filters (13) and reach said pneumatic system (15), from which they are collected in vessel (8b) and transferred to container (6), as described above.

Another important aspect of the invention. which is considered to be a great advantage. is the composition of the gases when they leave the melting oven (5). Typically, the composition of said gases contain only small amounts of oxides of nitrogen. sulfur and vanadium. and therefore fuel which is rich of

sulfur and/or vanadium may be used. As can be appreciated by one skilled in the art, this comprises both economical and ecological advantages, since these gases may be further used in other industrial applications.

Referring to Fig.2, According to another embodiment of the invention, the energy use is improved by using heat exchanger of cyclone type. Numeral (6) indicates a drying oven or the like, wherein said initial salt mixture is subjected to some small extant of heating to remove humidity. The mixture is collected in a container (5), and is then fed(4), in a controlled manner, into an ejector (3), and then transferred, by the use of an air stream (3a), to the cyclone heat exchanger. The oven for carrying out the melting is indicated by numeral (1), wherein burners(9) are provided. The salt mixture is passing through the cyclone heat exchanger(2) in the way to the oven, and the gases produced during the melting are driven into the heat exchanger, where they flow in a direction opposite to that of the air and salt, thus heating said salt mixture, in their way to the drying oven and from said drying oven to mechanical filters(7). Salt that was carried over by the gases is collected at the filters and transferred (8) to the oven. The molten salt mixture obtained at the oven is transferred (9) to the granulation process as described above.

Referring to Fig. 3, the drum is illustrated. The surface of the drum (1) is provided with recesses (2), dimension of which is substantially of the granules to be formed. Numeral (3) indicates the internal water cooling.

Referring to Fig. 4, a preferred embodiment of the drum is illustrated schematically (the view is of the rotary axis). The drum (1) coupled to a roller (2), their axes being parallel and they touch each other along a line. When the

rotational movement of the drum is taking place (3), the roller is performing an opposite rotational movement (4) by which material that was solidified on the surface of the drum is removed, the upper face of the granules in the recesses are leveled and the granules are brought into a uniform shape.

Example 1

Table I summarizes the results of the granulation process according to the embodiment of Fig. 1, for various initial compositions and conditions(i.e. dimensions of recesses in the drum). Initial KCl and K_2SO_4 were obtained from Dead Sea Works Ltd., in the form of a powder. The amount of mazout needed is about 42 kg per tone of salt.

initial KCl (kg)	initial K_2SO_4 (kg)	total weight of final granules (kg)	dimension of final granules (mm)	compression load (kg)
50.0	1.5	51.5	3.5-4.5	5.0-7.0
50.0	0.75	50.75	3.5-4.5	4.0-6.0
50.0	0.75	50.75	2.0-3.0	3.0-4.5

It will be apparent that the above-described apparatuses show only some preferred embodiments of the invention and that it is possible to carry out the invention by using different means for feeding the initial salt mixture, various kinds of ovens to melt said mixture, etc. It is surprising that, as long as the initial mixture comprises an addition of K_2SO_4, as set forth hereinbefore, and the cooling of the molten drops is carried out as set forth hereinbefore, the granules thus obtained have properties, in particular compressive strength, not attainable by any means known in the art.

Claims

1. A process for producing granulated potassium chloride, comprising:
- forming a mixture of potassium sulfate and potassium chloride;
- melting said mixture;
- bringing said molten mixture into physical contact with a cooling surface provided with recesses, said recesses being substantially of dimension and shape of the granules to be produced, said surface being cooled, whereby solidification of the molten mixture is obtained; and
-collecting the solid granules from the said surface.

2. A process according to claim 1, wherein melting is carried out in an oven, preferably a cyclone oven.

3. A process according to claim 1, wherein the cooling surface is made of material possessing high thermal conductivity.

4. A process according to claim 1, wherein the cooling surface is cooled by a cooling fluid.

5. A process according to claim 4, wherein the cooling fluid is water.

6. A process according to claim 1, wherein the granules are further cooled and the heat removed therefrom is recycled.

7. A process according to any one of claims 1 to 6, wherein the salt mixture contains about 1 to 5% by weight K_2SO_4 and 99 to 95% by weight KCl.

8. A process according to any one of claims 1 to 6, wherein the granules obtained from the solidification of the molten drops have size comprised between about 1.5 and 6 mm.

9. A process according to any one of claims 1 to 6, wherein the granules obtained from the solidification of the molten drops have a compressive strength of about 3 to 7 kg.

10. A process according to claim 1, wherein the cooling surface is provided on a drum capable of making rotational movement.

11. A process according to claim 10, wherein the drum is made of iron and is optionally coated with a removable porous coating.

12. A process according to any one of claims 10 or 11, wherein the drum is coupled to a roller to break salt that was solidified out of the recesses and thus to level the upper face of the granules.

13. A process according to any one of claims 10 to 12, wherein granules are dropped from the surface during the rotational movement of the drum to a container, and further cooled by means of heat exchanger, and the heat is recycled.

14. A process according to any one of claims 1 to 13, wherein the salt mixture is fed to the oven by the gases that are produced during the combustion of hot fuel and hot air.

15. A process according to claim 14, wherein the gases that flow out of the oven are in temperature of about 900°C-950°C.

16. A process according to claim 14, wherein the fuel is mazout or gas.

17. A process according to claim 15, wherein the fuel is sulfur rich.

18. A process according to claim 16, wherein the fuel is vanadium rich.

19. A process according to any one of claims 14 to 18, further comprising using the energy of the gases after the melting, said gases are finally brought to a lower temperature, typically in the range 200°C-250°C.

20. A process according to any one of claims 14 to 17, said gases, when leaving the melting oven. are almost free from oxides of sulfur and vanadium.

21.A process according to claim 1. wherein melting is carried out at a temperature of about 850°C.

22. A Process according to any one of claims 8 to 10. further comprising means for improving the process efficacy, whereby the gases used to drive the salt mixture to the oven, leave the oven and are brought into contact with heat exchangers, KCl-K_2SO_4 particles that were carried over by said gases are

formed as a result of the cooling of the suspension and are collected and transferred to the granulation system.

23. A process according to any one of claims 8 to 10, further comprising means for improving the process efficacy, whereby the gases used to drive the salt mixture to the oven, leave the oven and are brought into contact with heat exchangers, KCl-K_2SO_4 particles that were carried over by said gases are formed as a result of the cooling of the suspension and are collected and transferred to the granulation system, whereby the heat transferred from the gases is used to heat air from ambient temperature to about 450°C, said air is used as the hot air needed for the combustion that produces said gases.

24. A process according to any one of claims 8 to 10, further comprising means for improving the process efficacy, whereby the gases used to drive the salt mixture to the oven. leave the oven and are brought into contact with heat exchangers, KCl-K_2SO_4 particles that were carried over by said gases are formed as a result of the cooling of the suspension and are collected and transferred to the granulation system, whereby the heat transferred from the gases is used to heat air from ambient temperature to about 450°C, said air is used as the hot air needed for the combustion that produces said gases, whereby the gases are further passed through filters, to collect salt that remained therein. said salt is then melted and transferred to the granulation system.

25. Apparatus for producing granulated potassium chloride. which comprises:
- means for forming a mixture of potassium sulfate and potassium chloride;
- means for bringing said mixture to an oven;

- means for melting said mixture in the oven;
- means for obtaining the crystals by bringing said molten mixture into a physical contact with a cooling surface provided with recesses, said recesses being substantially of dimension and shape of the granules to be produced, said surface being cooled, whereby solidification of the molten mixture is obtained; and
- means for collecting the solidified granules from the surface and further cooling them.

26. Apparatus according to claim 25, wherein the oven is a cyclone oven.

27. Apparatus according to any of claims 25 or 26, comprising means for recovering heat from the hot gases which include heat exchangers.

28. Apparatus according to claim 25, wherein cyclone heat exchanger is applied.

29. A process for producing granulated potassium chloride, substantially as described and illustrated.

30. Apparatus for producing granulated potassium chloride, substantially as described and illustrated.

31. A process according to claim 29, characterized by an improved energy use, substantially as described in the specification.

32. A process according to claim 29, which is ecologically advantageous, substantially as described in the specification.

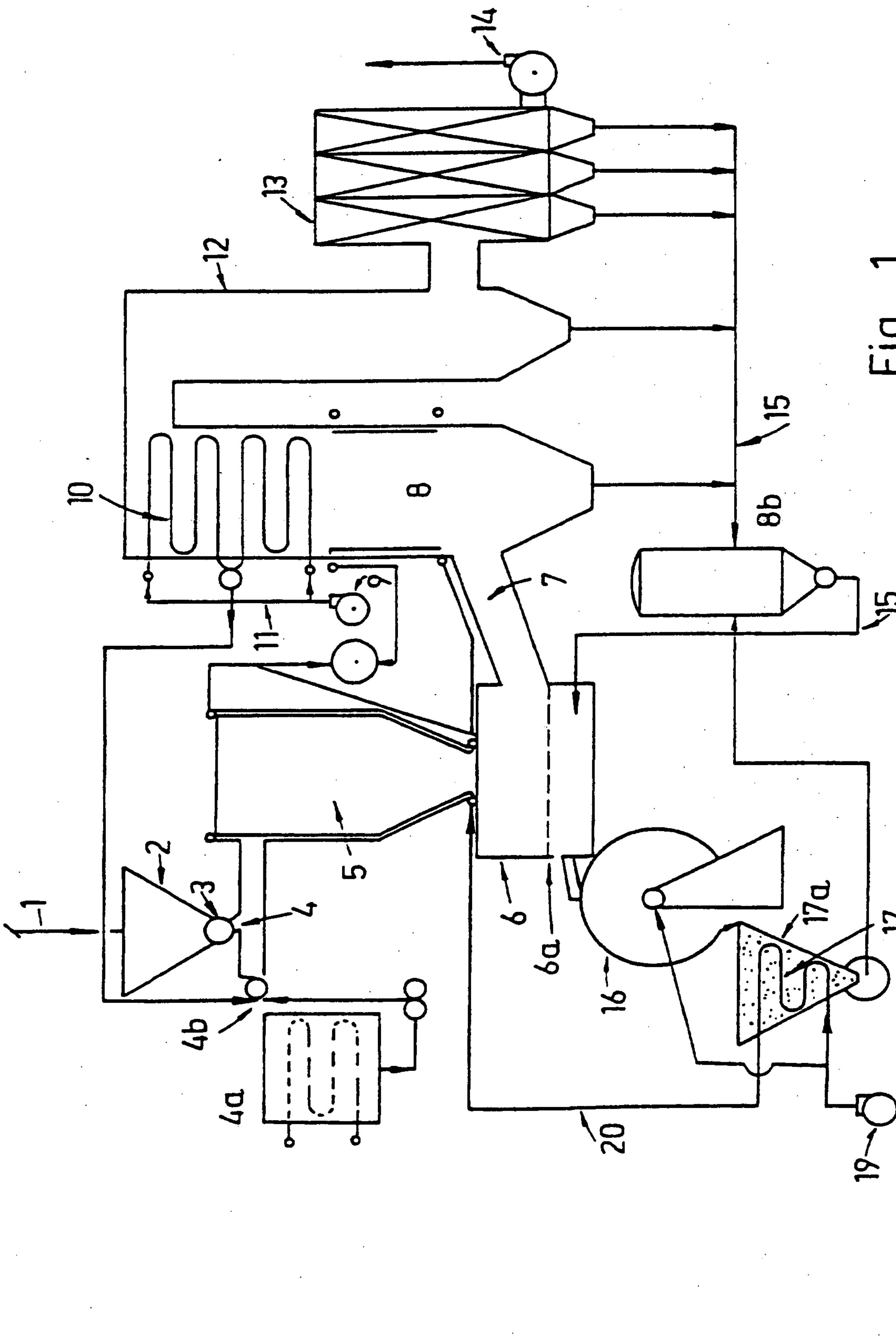

Fig. 1

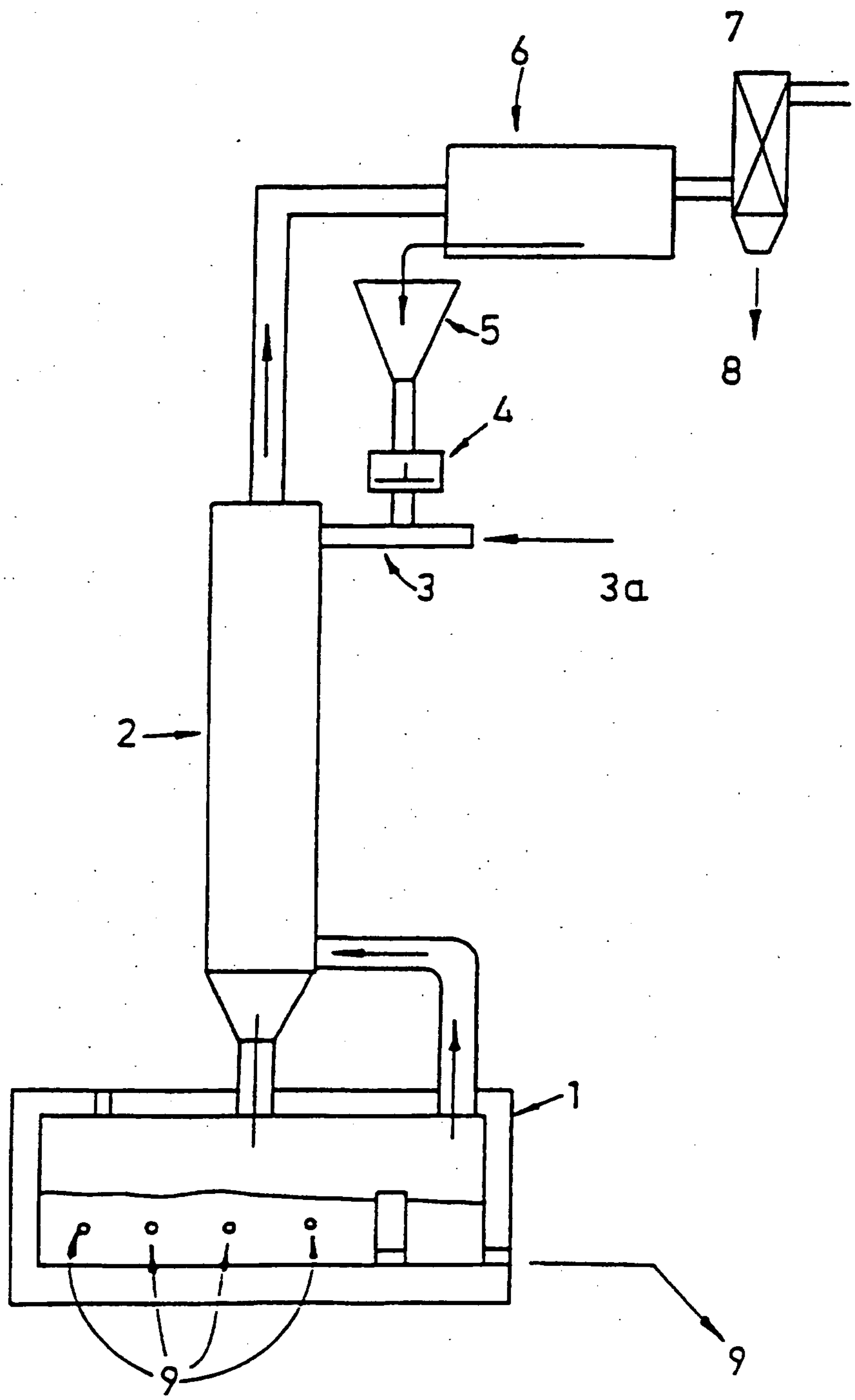

Fig. 2

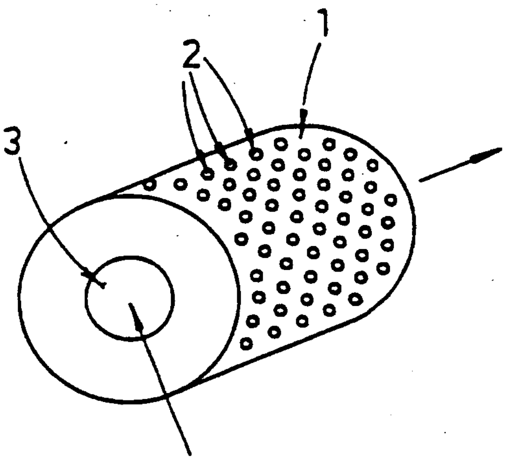

Fig. 3

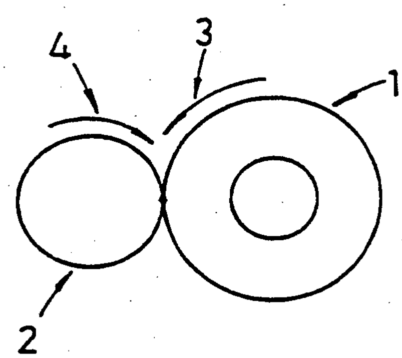

Fig. 4

INTERNATIONAL SEARCH REPORT

International Application No
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A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 C01D3/22 C05D1/02 B01J2/00 B01J2/22

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 C01D C05D C05G B01J

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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A	DATABASE WPI Week 79 Derwent Publications Ltd., London, GB; AN 82072 XP002007231 & SU,A,648 517 (P. V. KLASSEN) , 25 February 1979 see abstract --- -/--	1,7,9,25

[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	DATABASE WPI Week 71 Derwent Publications Ltd., London, GB; AN 12933 XP002007232 & JP,A,46 006 362 (MITSUI TOATSU CHEMICAL INC.) , 1971 see abstract ---	1,25
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Information on patent family members

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